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Work Plan for Chemically Enhanced Steam Stripping of Radionuclides in RFP Soils

Rocky Flats Plant

U.S. Department of Energy
Rocky Flats Plant
Golden, Colorado

Environmental Restoration Program

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Title: Work Plan for Chemically Enhanced Steam Stripping of Radionuclides from RFP Soils

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Table of Acronyms

ACWP	Actual Cost of Work Performed for the Month
Am	Americium
AO	Albuquerque Office
B/S	Bench Scale
BCWP	Budgeted Cost of Work Performed for the Month
BCWS	Budgeted Cost of Work Scheduled for the Month
CAR	Corrective Action Report
CCl_4	Carbon Tetrachloride
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CESS	Chemically Enhanced Steam Stripping
Ci	Curie
cm	centimeter
COC	Chain of Custody
CST	Chemical and Separations Technology
DNAPLs	Dense Non-Aqueous Phase Liquids
DOE	Department of Energy
dpm	disintegrations per minute
DQOs	Data Quality Objectives
DUGS	Dynamic Underground Stripping
EPA	Environmental Protection Agency
EQS	Environmental Quality Support
ER	Environmental Restoration
ERM	Environmental Restoration Management
ES&E	Environmental Science and Engineering
FY	Fiscal Year
g	gram
H&S	Health and Safety
HSP	Health and Safety Plan
IAG	InterAgency Agreement
IHSS	Individual Hazardous Substance Sites
IM/IRA	Interim Measure/Interim Remedial Action
INEL	Idaho National Engineering Laboratories
LA	Los Alamos
LANL	Los Alamos National Laboratory
LATO	Los Alamos Technology Office
lbs	pounds
LCS	Laboratory Control Sample
LLNL	Lawrence Livermore National Laboratories
LLW	Low Level Waste
M	Molar
M&TE	Measuring and Test Equipment
MDA	Method Detection Limit
MDA	Minimum Detectable Activities
mm	millimeters

Table of Acronyms (continued)

MSC	Measured Concentration in the QC check sample
MSDS	Material Safety Data Sheet
NAPLs	Non-Aqueous Phase Liquids
nCi	nano-Curies
NCR	NonConformance Report
NTS	Nevada Test Site
NVOCs	Non-Volatile Organic Compounds
OU	Operational Unit
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCE	Tetrachloroethene
pCi	pico-Curies
PEEK	Poly-ether-ether-ketone
psi	Pounds per Square Inch
PRG	Preliminary Remediation Goal
Pu	Plutonium
QA	Quality Assurance
QAA	Quality Assurance Addendum
QAMS/005/80	Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans
QAPD	Quality Assurance Program Description
QAPjP	Quality Assurance Project Plan
QC	Quality Control
RFO	Rocky Flats Office
RFP	Rocky Flats Plant
RPD	Relative Percent Difference
SNS	Scientific Notebook System
SRM	Standard Reference Material
TA	Technical Area
TAC	True Analyte Concentration in the QC check sample
TCE	Trichloroethylene
TD	Technology Development
TTP	Technical Task Plan
um	micron
VISITT	Vendor Information Service for Innovative Treatment Technologies
VOC	Volatile Organic Compound
°C	Degrees Celsius
%C	Percent Completeness
σ	Standard Deviation / sigma

1.0 Project Description

1.1 Introduction

Soils (and groundwater) contaminated with trace amounts of plutonium, americium, uranium mixed with volatile organic compounds (VOCs) and dense non-aqueous phase liquids (NAPLs) remain lingering problems in many remediation programs. Large volumes of contaminated soils may require processing to reduce contaminants to acceptable (yet undefined) levels.

At the Rocky Flats Plant (RFP) site, there are several below-ground locations that are contaminated with organic compounds and radionuclides (plutonium, americium, uranium). The particular focus in this study is Operational Unit (OU) 2/903 Pad area which contains radionuclides (plutonium, americium, and uranium), dense non-aqueous phase liquids (DNAPLs) which contain VOCs: trichloroethylene (TCE), carbon tetrachloride (CCl_4), tetrachloroethene (PCE), chloroform, methylene chloride; and non-volatile organic compounds (NVOCs): cutting oils and lathe coolants. The permeability of these areas may permit the percolation of these contaminant plumes and subsequent migration and eventual discharge of these contaminants as seeps into the surface waters. Mitigation and/or remediation of non-radionuclide contaminant sources are described in the OU2 Subsurface IM/IRA.

This proposed work will evaluate the use of thermally enhanced aqueous extraction processing of soils for the removal of radionuclides. The concept marries the technologies of redox, chelation, and steam processing to meet the challenge of RFP's OU2/903 Pad Area soils. The project will conduct proof-of-principle, bench-scale evaluations of chemically enhanced steam stripping for the mobilization and removal of radioactively-contaminated soils. This effort is part of the treatability studies for remedial screening and selection process for actinide contaminated soils in OU2's 903 Pad Area, but has applicability to other radionuclide contaminated sites such as OU9.

Testing will be conducted in three tasks: (1) select promising chelator-redox systems using fast-turnaround, lab-scale, chemically enhanced steam extraction tests, (2) further refining the list of chemical enhancers to the more promising chelator-redox agent systems using bench-scale, soil-column washing tests, and (3) optimize the most promising chelator-redox systems using parametric bench-scale, soil-column washing tests. The objectives will be:

- To *select* appropriate chelating/redox-agent systems, and to define test matrix and work plan for bench-scale tests.
- To perform bench-scale tests to *evaluate* plutonium and americium mobilization/removal efficiency using suitable chelating agents and redox conditions.
- To perform bench-scale tests to *optimize* plutonium and americium removal using suitable chelating and redox conditions that minimize chemical loading and modification of soils.

The test plan to achieve these objectives flows as:

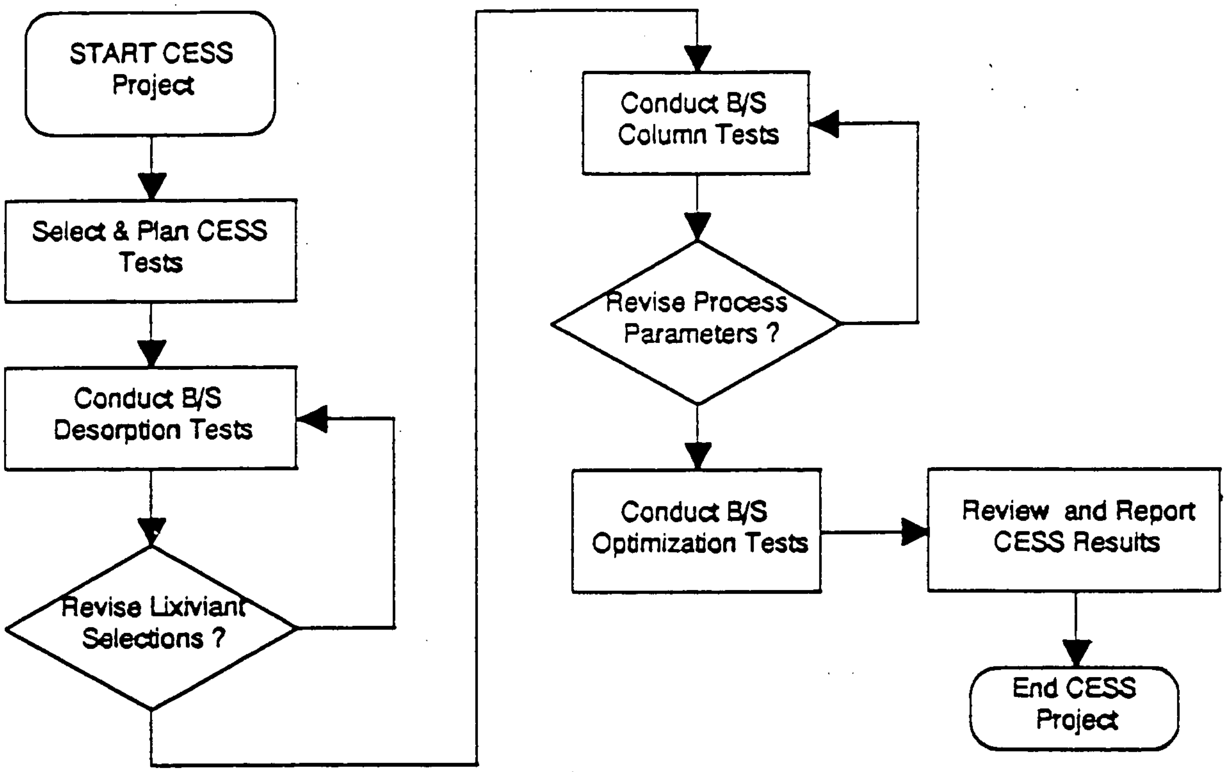


Figure 1. Test Plan for the CESS Project

1.2 Site History

Treatability studies will be performed on soils from the 903 Pad. The 903 Pad Area, encompassing the original 903 Drum Storage Site, was used from October 1958 to January 1967 for storage of radioactively contaminated oil drums whose contents were described by Calkins (1970).

"Most of the drums transferred to the field were nominal 55-gallon drums, but a significant number were 30-gallon drums. Not all were completely full. Approximately three-fourths of the drums were plutonium-contaminated, while most of the balance contained uranium. Of those containing plutonium, most were lathe coolant consisting of a straight-chain hydrocarbon mineral oil (Shell Vitrea) and carbon tetrachloride in varying proportions. Other liquids were involved, however, including hydraulic oils, vacuum pump oil, trichloroethylene, perchloroethylene, silicone oils, acetone still bottoms, etc...and in 1959 or possibly earlier ethanolamine was added to the oil to reduce the corrosion rate of the steel drums."

An estimated 5,000 gallons of liquid (Freiberg, 1970) containing 86 g (5.3 Ci) of plutonium leaked into the soil. Site grading in preparation for applying an asphalt cap over the area included moving "slightly" contaminated soil. A total of 33 drums of radioactively contaminated rocks were removed, and two courses of clean fill material were placed over the site. The asphalt covering was applied some two months later (Freiberg, 1970). The cover is approximately 8 centimeters (cm) thick and underlain by approximately 15 cm of loose gravel and 8 cm of fill dirt.

1.3 Project and Subtask Descriptions

The project will be conducted on 903 Pad area (sub)soils and in several phases comprising four subtasks. Strategic decision points, which allow judgment of the technology's promise and practice, are included in the project timeline.

Task 1. Bench-Scale Work Plan

This task covers the preparation of this work plan for bench-scale testing which follows "Suggested Organization of Treatability Study Work Plan" in EPA's *Guide on Conducting Treatability Studies under CERCLA* (EPA/540/R-92/071a) and ERM's procedure for "Work Plan Development".

This task will also include completing, as required, Health and Safety (H&S) plan and modification and revision of procedures for soil sampling and the handling of radioactive material prior to any work at RFP.

Task 2. Field Activities of the 903 Pad Area

This task will identify, based on past RFP OU2 work, and sample surface soils from locations which ensure sample representativeness. Soil will be collected and handled according to RFP procedures. The soil will be screened for radioactivity levels to ensure adherence to H&S radiological protection constraints before transferring to Los Alamos National Laboratory (LANL)/TA-48 for bench-scale tests.

Task 3. Bench-Scale Testing

Laboratory work will be performed at LANL but will be a significant collaboration between RFP and LANL. We will investigate the effectiveness of various redox and chelating agents that will mobilize plutonium and americium under steam conditions (near 80–100°C). Since plutonium solubility and chemistry varies with oxidation state, various reducing/oxidizing conditions will be evaluated for their impact on mobilizing plutonium. We will also assess the stability of chelating agents under steam conditions.

The details of the bench-scale testing, which will be conducted in three major subtasks, are described in the test plan provided in section 4 of this work plan.

Task 4. Conceptual Pilot-Scale Design (FY94)

This subtask will produce a conceptual pilot-scale design for a field operation. The design is anticipated to include chemically enhanced steam injection and associated feed and support subsystems, process control and monitoring technology, and any effluent collection and treatment subsystems.

2.0 Treatment Technology Description

2.1 Summary

There are various remediation approaches such as physical, chemical, biological, and thermal treatment, that can be used to remedy –whether by removal or stabilization– radionuclides and organics in soils and groundwater. Steam together with various types and concentrations of redox and chelating agents will be used to remove (i.e., mobilize and wash away) radionuclides contaminating selected RFP soils. Once radionuclides are mobilized, the radionuclide-rich (aqueous) mobile phase is driven by the steam front where it is collected and subsequently treated by more conventional wastewater technology. The approach uses a combination of steam injection and chemical action to mobilize radionuclides from soil where they can be subsequently treated by conventional technology.

2.2 Substrate/Soil Characterization

Retention of contaminants in a solid matrix (such as soils) varies with the physical and chemical properties of both the contaminant and the soil/solid phase. Contaminants are generally displaced by two mechanisms, either by: (1) physical action, driving or flushing the contaminant from the substrate, (2) chemical action, dissolving contaminant and/or substrate to release the contaminant, or by some combination of (1) and (2). Detailed knowledge of substrate/soil properties, chemistry, and nature of the substrate–contaminant interaction is extremely useful in predicting mobilization behavior, designing lixiviation systems, and shortening the process development time. Substrate characteristics are also important to predicting its response to chemical treatment.

Some information on contaminant and soil physical and chemical properties, which is useful in designing a treatability study, is available. Although recent soil characterization information (important because of the earlier excavation activity at the site) is not available, Hicks and Blakeslee (1981) reported limited soil physics and chemistry for samples collected from the 903 Pad area. Activity versus particle-size fractionation gave the following:

Table 2-1. Dry Screened 903 Pad Soils*

Fraction	> 4.0 mm	2.4 - 4.0 mm	0.42 - 2.4 mm	< 0.42 mm
Weight %	60	4	12	24
Pu (dpm/g)	240	1400	3100	29000
Am (dpm/g)	150	270	560	4100

*Soils dried at 100°C for 5 days. Data from Hicks and Blakeslee (1981).

Soil chemistries of selected, dried 903 Pad soils were also reported:

Table 2-2. Chemical Analysis of 903 Pad Soils (in wt %)**

Element	Al	Ca	C	Fe	K	Si	Na
Sample 1	5.4	0.9	0.8	1.7	2.5	30	1
Sample 2	5.2	3.0	1.4	2.1	2.3	21.5	1

** Data in weight percent; from Hicks and Blakeslee (1981).

This information should be used guardedly since recent studies suggest some mixing of the original and capping/fill materials has occurred. A study of selected physical and chemical characteristics of 903 Pad soils is currently being completed under the RFP ER program (Litaor, 1994a).

2.3 Soil Washing

EPA's Eagle et al. (1993) have demonstrated a soil washing plant for the treatment of radioactively contaminated soils at two Superfund sites. Soil washing operations were evaluated for their performance in reducing the volume of contaminated soils. Their program used a four-tiered approach consisting of: (1) soil characterization, (2) bench-scale testing, (3) process development units, (4) pilot plant development where the results of each tier were used to decide whether to proceed to the next tier.

2.4 Soil Washing at RFP

Soil decontamination and washing evaluations have been conducted at RFP since the early 1970s and results have appeared in reports in the internal and external literature. R. L. Olsen et al. (1980) described the decontamination of the 903 Pad area following cleanup of the leaking drums in 1968. They reported that radiological contamination of 2000-300,000 dpm/100 cm^2 had penetrated the 903 Pad soils to a depth of 20 cm.

Hicks and Blakeslee (1981) summarized a decade of soil characterization and bench-scale attrition scrubbing studies performed at RFP on RFP 903 Pad Area soils. Plutonium in these soils occurs in both particulate (0.2 micron (um) mean diameter associated with soil particles) and dissolved (or perhaps colloidal) forms. Wet screening and radiometric characterization of the soils showed that a majority of plutonium (and americium) was associated with soils of the 2.4 mm-and-less size fraction. Attrition scrubbing of soils with hot (80°C) distilled water or aqueous solutions of: chemical agents (e.g., H_2O_2, Na_2CO_3, NaClO, Na_2SiO_3), chelators (e.g., oxalic acid, citric acid), detergents (e.g., Oakite NST), and surfactants (e.g., sodium dioctyl sulfosuccinate) showed varying decontaminating effectiveness. Oxalic acid (0.1 molar (M)), sodium hexametaphosphate (10%), and 10% detergent solutions were among the more effective decontaminating systems for the 2.4-4.0 mm soil fraction, removing 95-98% of the Pu and Am. However, residual contamination levels still exceeded 60 dpm/g (27 pCi/g) following the scrubbing process. Hicks and Blakeslee (1981) also reported soil washing tests on soils from five DOE sites including RFP. Three aqueous solutions: (1) an aqueous pH 12.5, (2) 2% HNO_3, 0.2% HF, 2% pine oil, and 5% hexametaphosphate, (3) 2 Normal (N) HCl were evaluated for decontaminating RFP, Hanford, Mound, INEL, and LANL soils. Variability in the effectiveness of the three-phase scrubbing process was noted for soils from the different DOE sites. Effectiveness of the solutions also varied with the soil size fraction tested.

Pettis and Kallas (1988) conducted bench-scale testing and reported that simple, room temperature wet screening of 903 Pad soils was effective at decontaminating the greater than 4 mm size fraction (approximately 60 wt% of the total) to <5 dpm/g (2.3 pCi/g) Pu and Am. The >2.4 mm size fraction (approximately 65 wt% of the total) was decontaminated to less than 12 dpm/g (5.5 pCi/g) Pu and 6 dpm/g (2.7 pCi/g) Am by wet attrition scrubbing. The remaining, <2.4 mm soil fraction was treated by attrition scrubbing, ultrasonic scrubbing, oxidation, calcination, desliming, flotation, and heavy-liquid density separation. Although somewhat guarded because of results for selected size fractions, they concluded that attrition or vibratory scrubbing, and either mineral jig or acid leaching of this fraction would be effective for a decontamination goal of <30 dpm/g (14 pCi/g).

2.5 Steam Stripping

Of the various remediation approaches noted above, under the thermal category of remediation technologies, one innovative technology is dynamic underground stripping (DUGS) – which is an adaptation of steam injection and electrical heating. Steam injection accelerates removal of the NAPL contaminants and is combined with vacuum extraction to perform accelerated removal of volatile contaminants such as underground hydrocarbon spills and electrical heating accelerates the process. Steam injection technology has been demonstrated by Lawrence Livermore National Laboratory (LLNL) for remediating NAPLs and VOCs in subsurface soils and clay layers. Aines and Newmark (1992), and Buettner et al. (1992) (LLNL) have successfully tested this technology, in combination with electrical heating, on a bench scale and small field scale for the removal of NAPLs/VOCs in soils or clay layers at LLNL. EPA's Vendor Information Service for Innovative Treatment Technologies (VISITT) reports that this steam stripping technology is being commercialized by Praxis Environmental Services (San Francisco, CA) (Stewart, 1992).

2.6 Redox Chemistry

Mobilization of contaminants occurs as a result of physical or chemical action. Chemically induced mobilization occurs either by chemical action on the contaminant directly (e.g., plutonium) or its support substrate, in this case soil. In the case of plutonium-contaminated soils, the mobilization of soil-bound species depends to a large extent on the physical and chemical properties of both the plutonium and the soil.

Plutonium generally exists in four oxidation states: III, IV, V, and VI. In the natural environment, plutonium is normally found in either the IV state or, to a lesser extent, the V and VI states. The oxidation state of the plutonium is a determining factor in its solubility – III, V, and VI states being more soluble than the IV state, whereas, americium normally occurs only in the III state. Redox (or chemical reduction/oxidation) behavior of plutonium features highly in its propensity for dissolution, and modification of its oxidation state is an important tool in changing solubility. Cleveland (1971) has described conditions for both reduction and oxidation of Pu(IV) using a variety of chemical reagents. We will evaluate simple reducing and oxidizing agents and conditions to accelerate dissolution/mobilization of the plutonium (see Section 4.1.2).

2.7 Chelation Chemistry

Mobilization of metallic contaminants is greatly enhanced by formation of strong attachments with chemical binding agents via a process of chelation. Chelation chemistry and chelators (or chelates) are often used in association with metals to accelerate dissolution, and/or stabilize/maintain solubility by diminishing the tendency to readsorb or precipitate. Once resuspended or dissolved, the soluble species (in this case radionuclide-chelate complexes) are stabilized by their association with the chelate, which in turn enhances their continued mobility. A variety of complexing or chelating agents, including the well known EDTA, are commonly used for this purpose. Chelating agents can also enhance dissolution by tightly binding the radionuclide and preventing readsorption or precipitation. Chelating agents or chelators to be evaluated are discussed in Section 4.1.2.

2.8 Chemically Enhanced Steam Stripping

We propose to marry the technology of steam injection with concepts of redox and chelation for the leaching/washing of radionuclides from soils. Steam injection technology, enhanced by redox and chelation chemistry, will be used to mobilize and flush away radionuclides contaminating RFP soils.

The technology will be refined to minimize chemical injection with the goal of conducting ex situ treatment with the eventual return of the treated soils to the site. The benefits of this approach include: (1) reducing the volume of contaminated soils, (2) avoiding extremely harsh conditions therefore improving the potential for soil post-treatment soil viability and/or replacement, (3) possible tailoring to variable contaminants and soil conditions, and (4) combining with stream stripping of VOCs. The approach is also potentially applicable to in situ operations.

3.0 Test Objectives

Testing will be conducted in three tasks: (1) select promising chelator-redox systems using fast-turnaround, lab-scale, chemically enhanced steam extraction tests, (2) further refining the list of chemical enhancers to the more promising chelator-redox agent systems using bench-scale, soil-column washing tests, and (3) optimize the most promising chelator-redox systems using parametric bench-scale, soil-column washing tests. The objectives will be:

- To *select* appropriate chelating/redox-agent systems, and to define test matrix and work plan for bench-scale tests.
- To perform bench-scale tests to *evaluate* plutonium and americium mobilization/removal efficiency using suitable chelating agents and redox conditions.
- To perform bench-scale tests to *optimize* plutonium and americium removal using suitable chelating and redox conditions that minimize chemical loading and modification of soils.

Soil targets or decontamination levels are not available for this effort. The test plan will evaluate various chemical systems with the goal of diminishing Pu levels from a nominal value of 500-1000 pCi/g to as "low as technically practical," perhaps in the range of 5-50 pCi/g. Overall performance of washing solutions/conditions will be ranked based upon performance, including: effectiveness, efficiency, robustness, as well as other criteria including: cost and environmental acceptability.

4.0 Experimental Design and Procedures

4.1 Experimental Design

4.1.1 Background

Leaching or extraction agents were selected based on a combination of prior experience at RFP (Section 2.4), previous experimental results obtained by LANL with plutonium-contaminated soil from the Nevada Test Site (NTS), and recent results of soil washing of RFP at 20°C performed at LANL. NTS soil washing tests involved prescreening of soils by size fractionation of air-dried soil and subsequent alpha-particle counting of the different soil fractions to rank the size fractions according to their activity. The results indicated that most of the plutonium was associated with soil particles less than or equal to 53 microns in size. Consequently, all the leaching experiments with soil from the NTS were performed with a particle size of less than or equal to 53 um in size.

The experimental parameters of the NTS soil leaching experiments were: soil to solution ratio was 1:8, the contact time between the solution and solid phase was 24 hours, the concentration of all solutions was 0.1 molar (M), and the separation of phases was conducted by centrifugation. The initial Pu-239 concentration of the NTS soil was approximately 3000 dpm/g (1360 pCi/g) and all tests were conducted at 20°C. The results are given in Table 4-1. Although caution is appropriate in interpreting the results, Citric Acid and EGTA extractions gave promising results.

Table 4-1. Results of Plutonium-Leaching Experiments from the NTS Soils

Extractant	% Removal	Starting pH	Equilibrium pH
EDTA / 3% H_2O_2	22	4.4	7.7
EDTA	21	4.5	5.6
EDTA / 1 eq NaOH	14	8.0	8.6
EDTA / 2 eq NaOH	9	11.0	10.9
EDTA / 3 eq NaOH	5	12.3	12.0
Citric Acid / 3% H_2O_2	40	1.9	3.7
Citric Acid / 1 eq HNO_3	18	1.4	3.1
Citric Acid	16	2.2	3.8
Citric Acid / 1 eq NaOH	12	3.8	5.2
Citric Acid / 2 eq NaOH	10	5.1	8.2
Citric Acid / 3 eq NaOH	2	11.6	10.2
Citric Acid / 4 eq NaOH	0	12.3	12.3
Na_2EGTA	13	7.4	6.3
Na_2EGTA / 2 eq NaOH	45	12.0	11.5

Subsequently, work was also performed in the latter part of FY93 on soils form the RFP OU2/903 Pad Area. Draft results were reported in "Batch experiments for Desorption of Pu and Am in Contaminated Soils from RFP" completed January, 1994. In this report LANL tested 64 redox-chelating agent combinations for their effectiveness in leaching plutonium (Pu) and americium (Am) from RFP soils at 20°C.

Table 4-2. Selected Results of Leaching Experiments on RFP Soils at 20°C

Chelating Agent	Reducing/Chemical Agent	% Pu Removed	% Am Removed
0.1 M Citric Acid	2 equiv. NaOH	18	18
0.1 M EDTA	2 equiv. NaOH	8	8
0.1 M EDTA	3 equiv. NaOH	8	8
0.1 M DTPA	3 equiv. NaOH	24.5	24.5
0.1 M Sodium Citrate	0.01 M Fe(III) + 3% H_2O_2	43	43
0.1 M Citric Acid	3% H_2O_2	39	39
0.1 M Na_2CO_3	0.1 M Ascorbic Acid	20	20
0.1 M NTA	3 equiv. NaOH	22.5	22.5
0.1 M Sodium Citrate	0.1 M $Na_2S_2O_8$	28.5	28.5
0.1 M Sodium Citrate	0.1 M $Na_2S_2O_4$	60.5	60.5
0.1 M EDTA	3% H_2O_2	26	26
0.1 M Sodium Citrate	0.1 M $NH_2OH{\cdot}HCL$	36	36
0.1 M HNO_3	-	0	0
-	0.1 M Ascorbic Acid	0	0

Several promising candidate leaching systems were identified. The most efficient extractant was sodium citrate with almost any oxidizing or reducing agent tested. The overall best extraction of Pu and Am was with sodium citrate and the reducing agent, dithionite. Citric acid was also efficient as an extractant with most any additive, but it appears to be more efficient for Pu than for Am and no better than sodium citrate for Pu. Other complexants that show some promise are EDTA, especially with oxidizing agents, as well as DPTA and nitriloacetic acid. The results reported show some extraction efficiencies 50% or more.

Additional preliminary results are also available from sequential extraction of selected OU2 soils (Litaor, 1994b). Radioanalytical results from this work indicates that aqueous extractions using NaOCl and citrate-bicarbonate-dithionite were effective in mobilization of both Pu and Am. These and results mentioned above indicate that simple redox agents and complexants can desorb plutonium (and americium) from contaminated soils.

4.1.2 903 Pad Soil Testing Design Basis

The test matrix in Table 4-3 shows leaching experiments that were performed by LANL at 20°C under another project entitled, "Leachability of Pu from RFP Soils."

Table 4-3. Test Matrix for Plutonium-Leaching Experiments Using Standard Leaching Techniques

complexants (↓) redox agents (→)	water	NaOCl	NH_2OH-HCl	Dithionite
water		X	X	
Citrate/Bicarbonate				X
EDTA	X			
HNO_3	X			
$(NH_4)_2CO_3$	X			
Citric Acid	X			

Table 4-3 experiments tested simple complexants, standard leaching techniques, and redox behavior of the plutonium in RFP 903 Pad soil samples. Based on early results from similar experiments and knowledge of standard redox and solution behavior of plutonium (Cleveland, 1970), the leaching-test matrix shown in Table 4-4 was designed. Tests will be performed under steam conditions, about 80-90°C, on the RFP 903 Pad soil samples under the chemical conditions shown in Table 4-4.

Table 4-4. Test Matrix for Plutonium-Leaching Experiments Using Combination of Complexants and Redox Agents*

complexants (↓) redox agents (→)	water	NaOCl	H_2O_2	Ozone / Catalyst	Ascorbate	$NaHSO_3$/ H_2S
water					X	X
EDTA/Bicarbonate			X			
EGTA	X					
Carbonate / Bicarbonate	X	X	X	X	X	X

* As test results become available, other chelating and reducing agents may be added to the test matrix, as appropriate.

Other suitable chelating and redox agents may also be considered and tested under the steam conditions as the test results become available.

4.2 Test Plan

Technical testing efforts will be devoted to three main areas: desorption or leaching experiments, bench-scale column experiments, and bench-scale optimization, described below.

4.2.1 Batch Desorption Experiments

Leaching experiments involve taking soil from the Rocky Flats OU2, adding a solution containing a complexing agent, mixing the two phases, separating the phases, and determining the amount of plutonium and americium in each phase. These leaching experiments will be conducted at 80°C using the experimental set-up depicted in Figure 2. This set up consists of equilibrating tubes containing the contaminated soil and the leaching solution in a shaker surrounded by a metal enclosure that is kept at 80°C using a recirculating oil bath. The actual procedure to be utilized for desorbing plutonium and americium from the Rocky Flats soil will be TWS-INC-DP-05, R2 "Sorption, Desorption Ratio Determinations of Geologic Materials by a Batch Method," and LANL-INC-DP-86, R0 "Sorption and Desorption Determinations by a Batch Sample Technique for the Dynamic Transport Task." The determination of the amount of plutonium in each phase will be performed by alpha spectrometry following the procedures in LA-1721, 5th edition "Collected Radiochemical and Geochemical Procedures." The determination of the amount of americium in each phase will be performed by gamma spectrometry following the procedures in LA-1721, 5th edition "Collected Radiochemical and Geochemical Procedures." Each experiment will be performed in duplicate.

The choice of leaching agents was made taking into consideration results obtained: with plutonium-contaminated soil from the Nevada Test Site (NTS), from leaching Pu and Am from RFP soil at 20°C in 1993, and in previous studies as described in Section 4.1.

Table 4-5 presents 18 experiments (to be performed in duplicate) at 80°C. The concentration of the complexants in the leaching solutions to be utilized will be approx. 0.1 M. The concentration of the redox agents in the leaching solutions will be approx. 30-50%. In the test matrix shown in Table 4-4, the vertical columns are the complexants and the horizontal rows are redox agents that will be used with the indicated (X) complexants. Other experimental parameters are as follows. The soil to solution ratio will be 1:10, the contact time between the solution and solid phase will be 24 hours, and the separation of phases will be conducted by centrifugation.

Figure 2. Equipment for Batch Desorption Experiments at 80°C.

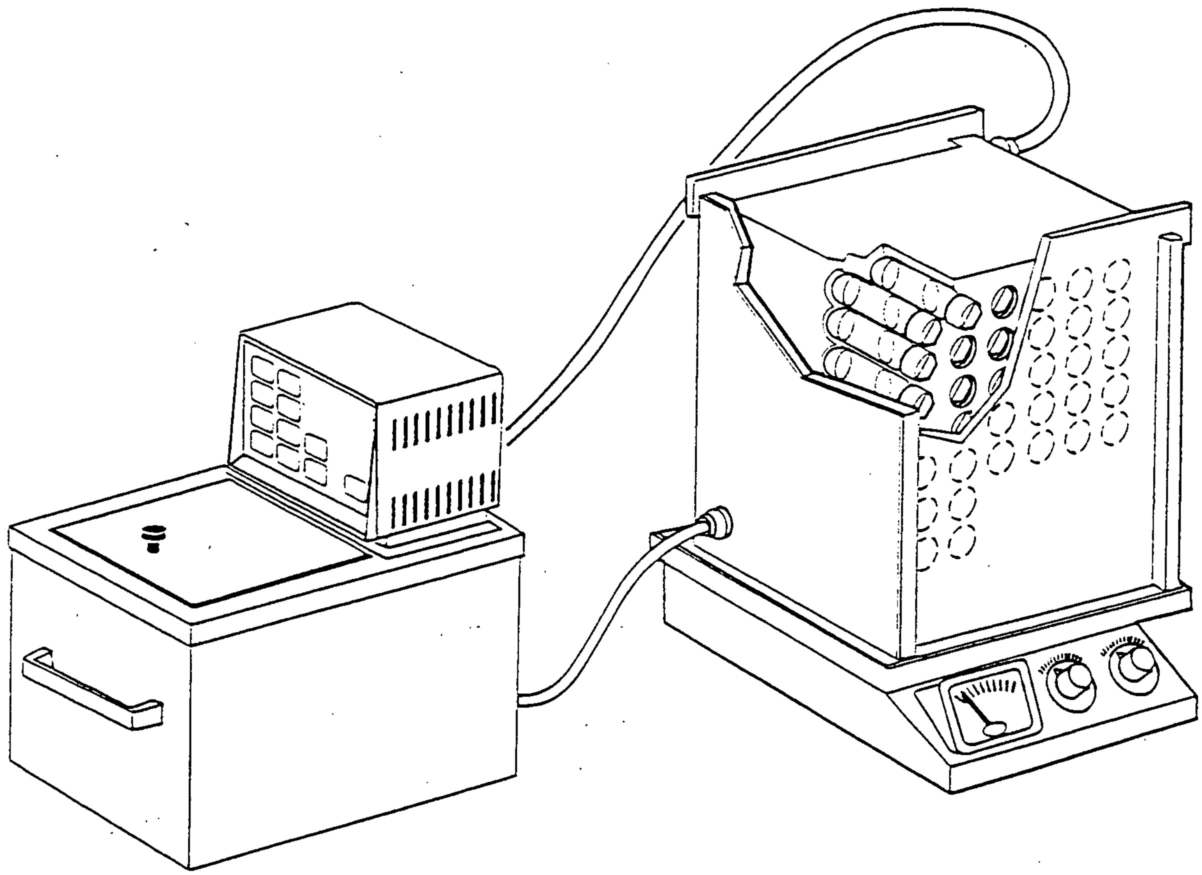


Table 4-5. Test Matrix for Plutonium and Americium Leaching Experiments Using Combination of Complexants and Redox Agents

complexants (↓) redox agents (→)	water	NaOCl	H_2O_2	Ascorbate	Dithionite
water		X	X	X	X
Sodium citrate	X	X	X	X	X
EDTA	X		X		X
Nitrotrilotriacetic acid	X		X		X
DTPA	X		X		X

4.2.2 Bench-Scale Column Experiments

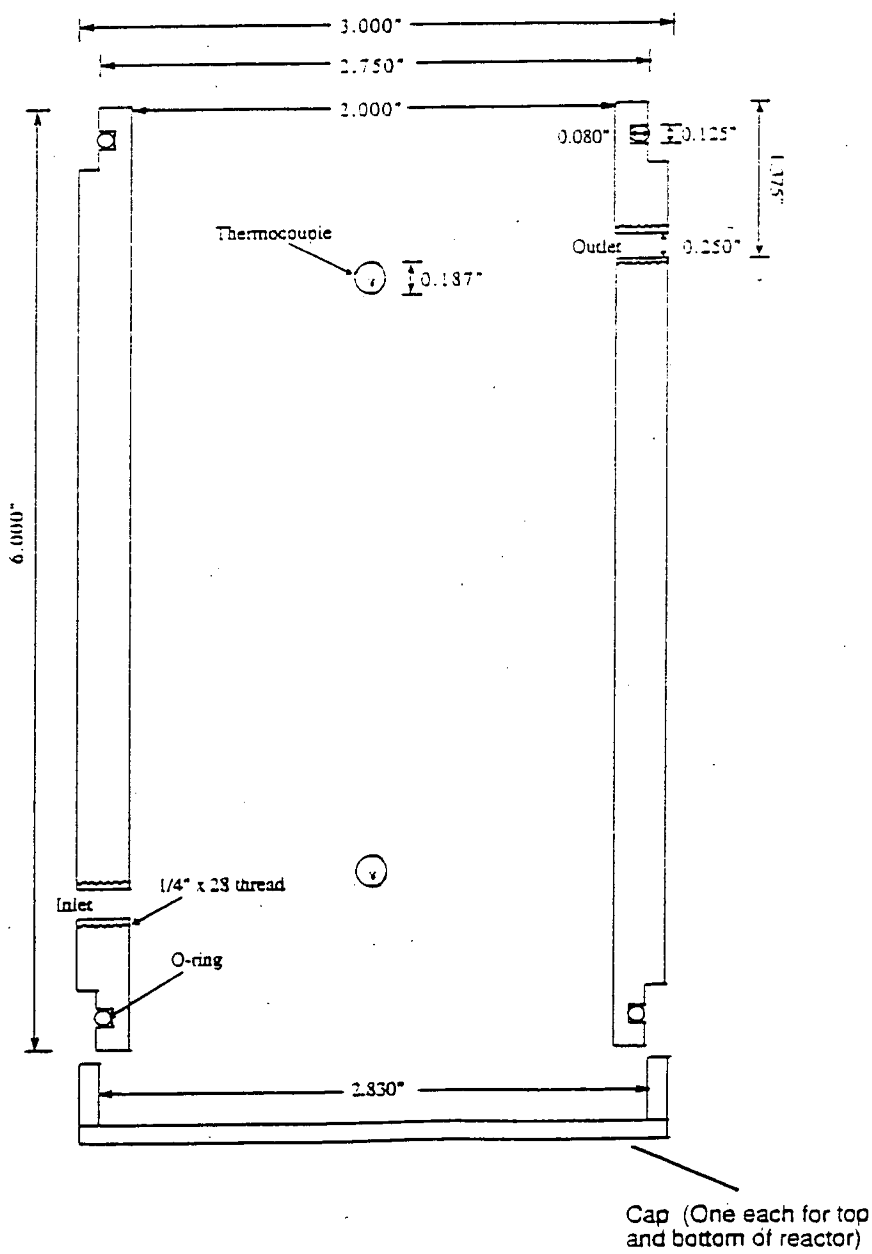
Based on results of batch experiments, additional tests will be conducted using the more promising solutions which will be eluted at 80°C through heated columns containing RFP soils. The procedure to be utilized for the column experiments is given in LANL-INC-DP-i5, R3 "Crushed Rock Column Studies." Tests will be conducted in small-volume columns — containing 25g or more of RFP soil — to maximize the number of extractant systems that can be evaluated. Ten to 20 column volumes will be eluted to determine the amount of plutonium and americium leached from the soil. The column experiments will be performed in duplicate, and the amount of plutonium and americium leached from the RFP soil will be determined by alpha and gamma spectrometry, respectively, following the procedures in LA-1721, 5th edition "Collected Radiochemical and Geochemical Procedures."

4.2.3 Bench-Scale Optimization Studies

The optimal leaching solutions will be carried forward from the results of the bench scale column studies, and used to refine the soil washing parameters for the chemically enhanced steam stripping process. The studies in the laboratory-scale optimization will entail maximizing the amount of plutonium and americium leached from the RFP soil while minimizing the lixiviant or solution strength. The equipment configuration to be utilized is depicted in Figure 3. Selected steam-activated leaching solutions will be injected into a reactor containing 200 g of contaminated RFP soil. The reactor will have an approximate internal diameter of 2 inches and a height of 6 inches and be made of inert materials such as the commercial polymeric material, PEEK[1]. Several reaction vessels will be fabricated to sustain a working pressure of 100 psi. The plutonium and americium concentration in each phase will be determined by alpha and gamma spectrometry, respectively, following the procedures in LA-1721, 5th edition "Collected Radiochemical and Geochemical Procedures." A careful mass balance of the plutonium and americium concentration in each phase will be performed in this effort.

[1] Poly-ether-ether-ketone (PEEK)

Figure 3. Bench-Scale Reactor for Plutonium Steam Stripping.



(Not To Scale)

5.0 Equipment and Materials

The desorption experiments require:

- Containers with leak-proof caps for sample containment
- A shaker kept at 80°C as depicted in Figure 2
- Ultra and super speed centrifuges to separate the solid and liquid phases after the leaching experiments are completed
- Calibrated balance
- Standardized counters to determine the amount of plutonium in each phase after leaching. (The type of counters used are determined by the LANL Procedure entitled, "Sorption, Desorption Ratio Determinations of Geologic Materials by a Batch Method," (TWS-INC-DP-05,R2, July 23, 1990).

The column experiments require:

- Columns
- Syringe Pumps
- Calibrated balance
- Rheodyne injection valve
- Poly-ether-ether-ketone (PEEK) or teflon tubing
- Fraction collector
- Standardized counters to determine the amount of plutonium in each phase after leaching. (The type of counters used are determined by the LANL Procedure entitled, "Sorption, Desorption Ratio Determinations of Geologic Materials by a Batch Method," (TWS-INC-DP-05,R2, July 23, 1990).

The optimization experiments require:

- A reactor as depicted in Figure 3 whose column is made out of PEEK
- Standardized counters to determine the amount of plutonium in each phase after leaching. (The type of counters used are determined by the LANL Procedure entitled, "Sorption, Desorption Ratio Determinations of Geologic Materials by a Batch Method," (TWS-INC-DP-05,R2, July 23, 1990).

6.0 Sampling and Analysis

Soil and other environmental materials from the sampling area will be handled according to procedures in the EMD Operating Procedures Manual (5-21000-OPS-FO, Rev. 51, 7/26/93). All RFP soils handled by LANL will be stored and handled according to TWS-INC-DP-83, R1 "Storage and Handling of Solid Samples."

Representative sampling of bulk soils for radionuclide analyses is difficult to achieve without some pre-treatment by physical methods. Reproducibility of analytical methods is also complicated by heterogeneities in the soil matrix. To minimize this experimental difficulty and maximize information specific to the chemical/steam conditions, we will dry and sieve the soils prior to all bench-scale experiments. As with previous studies (where plutonium is associated with a particular size fraction in the RFP soils), we will select a soil size fraction for bench-scale testing, which both optimizes experimental logistics and performance, while maintaining sampling and analytical reproducibility.

7.0 Data Management

Data acquisition and management at RFP follow the general DOE Standard DOE-ER-STD-600192, Implementation Guide for Quality Assurance Programs for Basic and Applied Research, Criteria 4 and 6. Data and other experimental information, which is collected by RFP for the test program in Section 4, will be recorded and managed in accordance with draft ER procedure for "Scientific Notebooks" (2-G06-ER-ADM-05.10, Rev. 0). Additionally, all data must be submitted to the ERM records center in a timely manner and comply with the QAPjP and QAMS-005/80.

At LANL, the data from procedures in test plan (Section 4) will be entered and stored in electronic spreadsheets using Microsoft Excel. Hard copies of the spreadsheets which will include the data obtained and any procedural deviations will be signed by the technician performing the work and the principal investigator. Electronic copies of the spreadsheets will be stored on a hard disk drive, which is backed up on to an optical disk on a weekly basis.

8.0 Data Analysis and Interpretation

The results of the experiments in Section 4 will be analyzed following the guidelines given therein. Experiments will be performed (at least) in duplicate and simple statistical methods applied to screening and parametric data. Effectiveness of radionuclide removal will be assessed by alpha spectroscopy of the soil washing liquids and/or soils following treatment. Because of the relative facility with which solution samples may be reduced for radioassay, radiometric methods will be preferentially performed using washing solutions to establish efficiency; residual soil levels will be determined by difference. Radionuclide mass balance will be carefully checked in Section 4.2.2 and in the parametric tests given in Section 4.2.3.

Although Hayden et al. (1980) have provided some discussion, no soil decontamination or targets levels are available.[2] The test plan evaluates various chemical systems with a goal of the best that can be done. Further evaluation of soil washing solutions, not effectively mobilizing plutonium to produce low soil residuals will be discontinued. This approach will accelerate early screening and refinement of options prior to optimizing/parametric studies in Section 4.2.3. Overall performance of washing solutions/conditions will be ranked based upon: effectiveness, efficiency, robustness, as well as other criteria including cost and environmental acceptability.

9.0 Health and Safety

All health and safety aspects of the work to be performed as specified in Section 4 will follow the requirements of either the site-specific Health and Safety Plan (HSP) for RFP as modified by facility-specific requirements of the testing laboratory, or the Health and Safety Procedures of the Chemistry and Separations Technology (CST) Division at LANL. CST's Procedures include instructions for handling, storage, and disposal of radioactive and hazardous materials at Technical Area-48, Building RC-1 of the Los Alamos National Laboratory, where the work will be performed.

10.0 Residuals Management

Residuals and investigatory materials will be characterized by process knowledge or assay to establish handling requirements. Residuals will be managed at the investigator's facility. At RFP residuals are covered in EMD Procedures Manual (5-21000-OPS-FO, Rev. 51, 7/26/93) or according to building-specific requirements for waste disposition. Any work at RFP will follow the RFP Waste Minimization Plan which emphasizes not generating or minimizing the generation of low-level waste (LLW) and/or mixed wastes. The treatability goal will be to maximize leaching efficiency while using minimal chemical treatment.

[2] Recent risk-based recommendations, which are still undergoing review, indicate that a Pu-239 in soils target of 3.4 pCi/g satisfies the "one-in-a-million" additional risk.

11.0 Reports

Periodic reports will be provided both to the RFO ER office and to the RFP Project Manager according to the attached schedule. Monthly letter/progress reports with provide: summary of significant results, projection of accomplishments for the next period, and a review of issues and problems affecting schedule. The format for monthly reporting will be:

<u>MONTHLY REPORTING FORMAT</u>

Task Order Number:

Task Order Title:

Month and Year of the Status Report:

Award Date of Task Order:

Task Order Funding:

1) Initial Funding
2) Modification Funding (If applicable):
3) Revised Total Funding (If applicable):

Amount Incurred to Date:

Total Amount Invoiced To Date:

Date and Number of Last Invoice:

Percentage of Work Effort Completed:

Budgeted Cost of Work Performed for the Month (BCWP):

Actual Cost of Work Performed for the Month (ACWP):

Budgeted Cost of Work Scheduled for the Month (BCWS):

Cumulative Budgeted Cost of Work Performed:

Cumulative Cost of Work Performed:

Cumulative Cost to Date:

Calculated Variances:

Actual Cost vs. Budgeted Cost (BCWP-ACWP x 100/BCWP)

MONTH CUMULATIVE

Work Scheduled vs. Work Performed (BCWP-BCWS x 100/BCWS)

MONTH CUMULATIVE

Additional Comments:

The following format (after section 3.12 of "The Guidance for Conducting Treatability Studies under CERCLA" (EPA 1992)) will be used for reporting technical results.

TREATABILITY STUDY REPORTING FORMAT

1.0 ABSTRACT/EXECUTIVE SUMMARY

2.0 INTRODUCTION
- 2.1 Site Description
- 2.2 Waste Stream Description
- 2.3 Technology Introduction

3.0 TREATABILITY STUDY APPROACH
- 3.1 Test Objectives and Rationale
- 3.2 Treatment Technology Description
  - 3.2.1 Treatment process and scale
  - 3.2.2 Operating features
- 3.3 Experimental Design and Procedures
- 3.4 Equipment and Materials
- 3.5 Sampling and Analysis
  - 3.5.1 Waste stream
  - 3.5.2 Treatment process
- 3.6 Data management
- 3.7 Deviations from the Work Plan

4.0 RESULTS AND DISCUSSION
- 4.1 Data Analysis and Interpretation
  - 4.1.1 Analysis of Waste Stream Characteristics
  - 4.1.2 Analysis of Treatability Study Data
  - 4.1.3 Comparison to Test Objectives
- 4.2 Costs/Schedule for Performing the Treatability Study

5.0 CONCLUSIONS AND RECOMMENDATIONS
- 5.1 Conclusions
- 5.2 Recommendations

6.0 Quality Assurance/Quality Control (QA/QC)

7.0 Key Contacts

References

Appendices
- A. Data Sheets, Lab Results, MSDSs
- B. Data Summaries
- C. Procedures

12.0 Schedule

Activities described under this work plan cover a two-year effort containing several decision points. If successfully shown at proof-of-principle and pilot-scale stages, field demonstration of this chemically enhanced steam technology could occur in 3-5 years. Activities planned for the near-term treatability testing of this approach are:

Task/Milestone Summary: FY 93	*Date Complete (mo.-day-yr.)*
a. Complete work plan/test plan	4-18-94
b. Initiate bench-scale tests	7-13-94
c. Issue Phase I Report	12-7-94
d. Issue Final Report	11-9-95

Milestone Explanation:

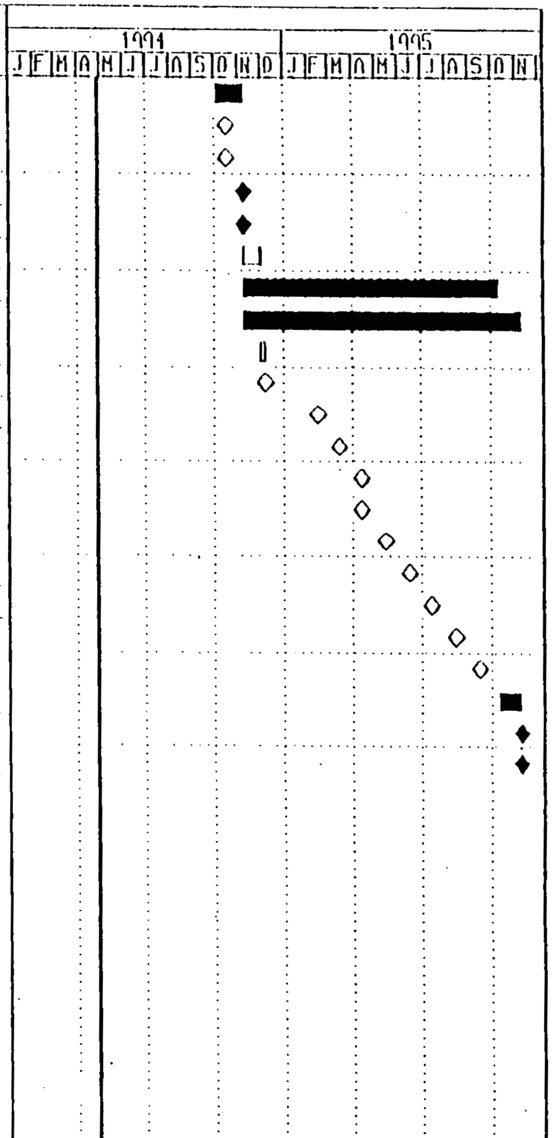
a. The test plan is contained within this work plan.

b. Start of actual lab testing, Phase I. Collection and delivery of RFP contaminated soil samples to LANL. This includes the selection, collection, and shipment to LANL of soil samples from the 903 Pad area.

c. Interim Report - A technical report describing bench-scale results and interim conclusions will provide sufficient details and information to substantiate continuing this effort to Phase II.

d. Final Report - A technical report describing results conclusions to Phase I and Phase II subprojects.

ACTIVITY ID	ACTIVITY DESCRIPTION	REM DUR	EARLY START	EARLY FINISH
CESS001250	CONT FY95 PHASE I TESTING	26	3OCT94	7NOV94
CESS001218	COMPLETE SEPTEMBER MONTHLY STATUS REPORT	0	17OCT94	
CESS001219	SUBMIT SEPT MONTHLY STATUS REPORT TO DOE/RFO	0	17OCT94	
CESS001235	START PHASE II TESTING	0		8NOV94
CESS001255	COMPLETE PHASE I TESTING	0		8NOV94
CESS001260	PREPARE FY94 FINAL REPORT	15	8NOV94	30NOV94
CESS001210	CONDUCT FY95 PHASE II ACTIVITIES	229	8NOV94	10OCT95
CESS001237	OVERSEE PHASE II TESTING	251	8NOV94	9NOV95
CESS001265	SCHEDULE RESERVE	5	1DEC94	7DEC94
CESS001270	COMPLETE FY94 CESS REPORT	0		7DEC94
CESS001274	COMPLETE MONTHLY STATUS REPORT	0	15FEB95	
CESS001275	COMPLETE MONTHLY STATUS REPORT	0	15MAR95	
CESS001276	COMPLETE MONTHLY STATUS REPORT	0	13APR95	
CESS001282	COMPLETE SEMI ANNUAL REPRT	0	13APR95	
CESS001277	COMPLETE MONTHLY STATUS REPORT	0	15MAY95	
CESS001278	COMPLETE MONTHLY STATUS REPORT	0	15JUN95	
CESS001279	COMPLETE MONTHLY STATUS REPORT	0	14JUL95	
CESS001280	COMPLETE MONTHLY STATUS REPORT	0	15AUG95	
CESS001281	COMPLETE MONTHLY STATUS REPORT	0	15SEP95	
CESS001290	PREPARE FINAL FY95 REPORT	21	11OCT95	8NOV95
CESS001300	SUBMIT FINAL CESS REPORT	0		9NOV95
CESS001310	COMPLETE CESS PROJECT	0		9NOV95



Plot Date 20APR94
Data Date 20APR94
Project Start 26JAN94
Project Finish 9NOV95

Activity Bar/Early Dates
Critical Activity
Progress Bar
Milestone/Flag Activity

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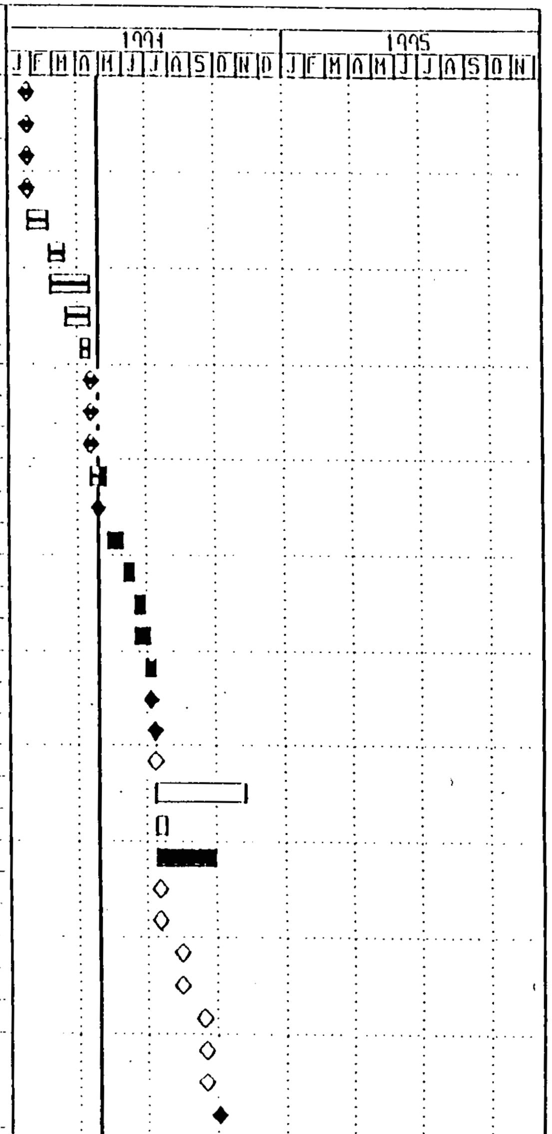
CESS

EG&G ROCKY FLATS
CHEMICALLY ENHANCED STEAM STRIPPING
OF RADIONUCLIDES FROM RFP SOILS

Sheet 2 of 2

Date	Revision	Checked	Approved

ACTIVITY ID	ACTIVITY DESCRIPTION	REM DUR	EARLY START	EARLY FINISH
CESS001000	START CESS PROJECT	0	26JAN94A	
CESS001010	START PROCUREMENT ACTIONS	0	26JAN94A	
CESS001020	START WORK PLAN REWRITE	0	26JAN94A	
CESS001030	START PROJECT MGT ACTIVITIES	0	26JAN94A	
CESS001050	REVISE CESS WP CONCEPT	0	26JAN94A	22FEB94A
CESS001040	REVISE CESS WORK PLAN	0	23FEB94A	15MAR94A
CESS001040	DEVELOP CESS SOW	0	25FEB94A	18APR94A
CESS001100	CONDUCT WP REVIEW & REVISION	0	16MAR94A	18APR94A
CESS001060	PREPARE SUPPORTING DOCUMENTS	0	5APR94A	18APR94A
CESS001110	START PROCMT CESS SERVICES	0		18APR94A
CESS001120	FINAL WORK PLAN COMPLETED	0		18APR94A
CESS001130	COMPLETE CESS SOW	0		18APR94A
CESS001140	PREPARE CESS RFP	7	19APR94A	9MAY94
CESS001125	SUBMIT FINAL WORK PLAN TO DOE/RFO	0		20APR94
CESS001150	PREPARE INTEGRATED CONTRACTOR PROPOSAL	15	10MAY94	31MAY94
CESS001160	COMPLETE CESS TECH EVAL	10	1JUN94	14JUN94
CESS001070	REVISE SAMPLING PROCED/GET PERMITS	10	15JUN94	28JUN94
CESS001155	SCHEDULE RESERVE	15	15JUN94	6JUL94
CESS001080	COLLECT & SHIP SOIL SAMPLES	10	29JUN94	13JUL94
CESS001170	CESS SUBCONTRACT AWARDED	0	7JUL94	
CESS001190	FY94 FUNDING AUTHORIZED	0	13JUL94	
CESS001195	START PHASE I TESTING	0	13JUL94	
CESS001180	OVERSEE PHASE I TESTING PROJECT	85	13JUL94	9NOV94
CESS001200	SUPP FY95 PROJ FUND & PROCMT	10	14JUL94	27JUL94
CESS001210	CONDUCT FY94 WP TESTING	56	14JUL94	30SEP94
CESS001211	COMPLETE JUNE MONTHLY STATUS REPORT	0	19JUL94	
CESS001212	SUBMIT JUNE MONTHLY STATUS REPORT TO DOE/RFO	0	19JUL94	
CESS001214	COMPLETE JULY MONTHLY STATUS REPORT	0	16AUG94	
CESS001215	SUBMIT JULY MONTHLY STATUS REPORT TO DOE/RFO	0	16AUG94	
CESS001230	FY95 SUBCONTRACT AUTHORIZED	0	14SEP94	
CESS001216	COMPLETE AUGUST MONTHLY STATUS REPORT	0	16SEP94	
CESS001217	SUBMIT AUGUST MONTHLY STATUS REPORT TO DOE/RFO	0	16SEP94	
CESS001220	CONTINUE FY95 PHASE I ACTIVITIES	0	30CT94	



Plot Date 20APR94
Data Date 20APR94
Project Start 26JAN94
Project Finish 9NOV95

Activity Bar/Early Dates
Critical Activity
Progress Bar
Milestone/Flag Activity

(c) Primavera Systems, Inc

CESS

Sheet 1 of 2

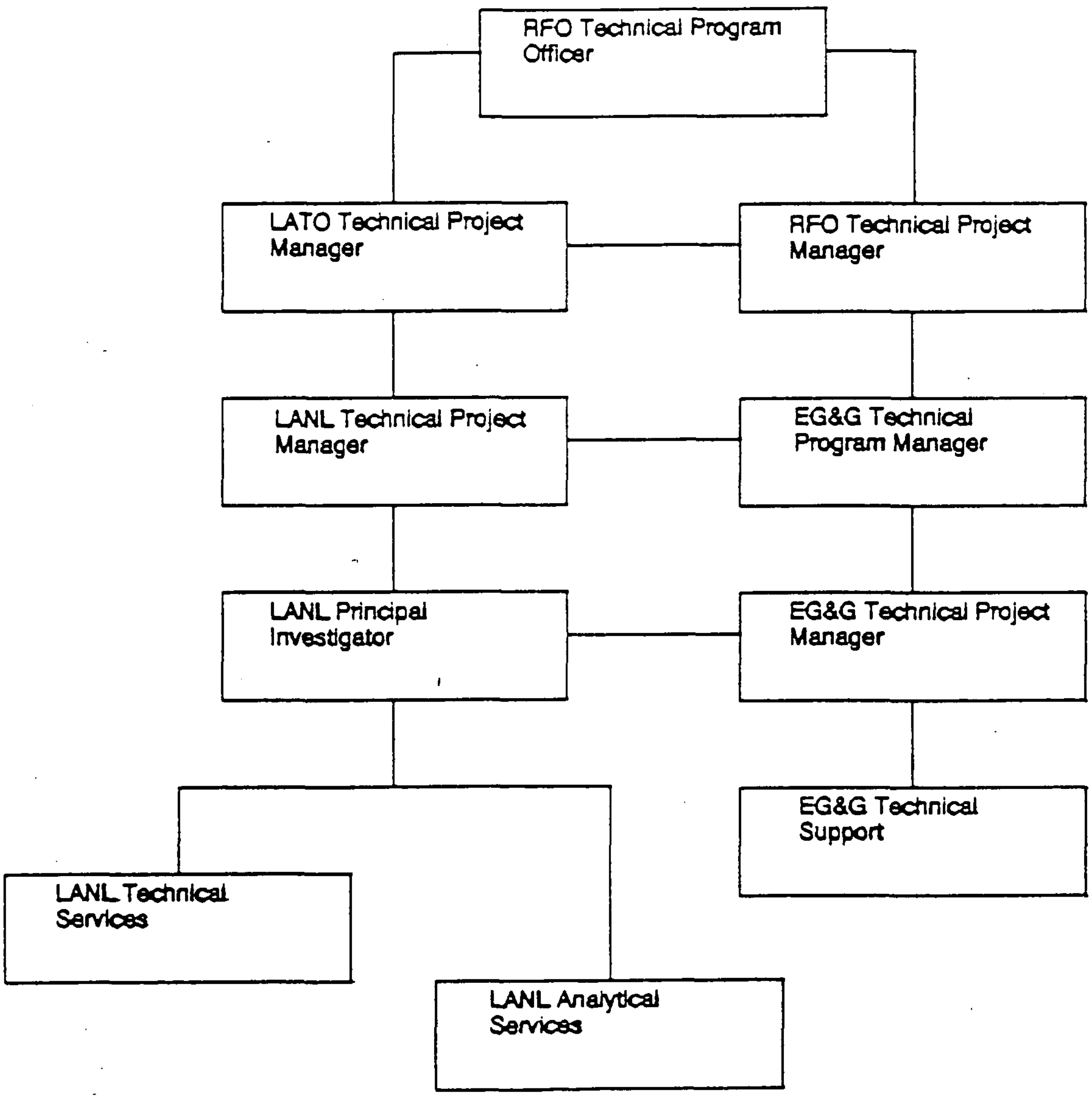
EG&G ROCKY FLATS
CHEMICALLY ENHANCED STEAM STRIPPING
OF RADIONUCLIDES FROM RFP SOILS

Date	Revision	Checked	Approved

13.0 Management and Staffing

This project is a joint effort among collaborators from RFP and LANL. The RFP Principal Investigator and Rocky Flats Office Project Manager will be the point contacts for the effort. The management and reporting structure is given in Figure 4.

Figure 4. Management and Reporting Structure



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M. Z. Litaor (b), personnel communication, March 21, 1994. Preliminary data from selected sequential extraction studies show citrate-bicarbonate-dithionite and NaOCl are effective extractors of selected OU2 soils.

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APPENDIX A

**Quality Assurance Addendum for the
Chemically Enhanced Steam Stripping Work Plan**

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1.0 PURPOSE

The purpose of the Quality Assurance Addendum (QAA) is to identify quality assurance (QA) requirements, and specific measures for implementing these requirements, that are applicable to the laboratory experiments addressing in situ chemical remediation of radionuclides in soil. This QAA is intended to supplement the "Rocky Flats Plant Site-Wide Quality Assurance Project Plan for CERCLA Remedial Investigation/Feasibility Studies and RCRA Facility Investigations/Corrective Measures Studies Activities" (referred to as the RFP Site-Wide QAPjP, or simply QAPjP). As a supplement to the QAPjP, this QAA establishes the specific measures and QA controls applicable to the actions described in this work plan. The purpose of this laboratory work is to characterize Pu and Am mobilization through soil within given redox conditions.

2.0 SCOPE

This QAA addresses all quality affecting activities described in the work plan to be performed by EG&G Rocky Flats; other organizations Los Alamos National Laboratory (LANL) and Lawrence Livermore National Laboratory (LLNL) implement similar QA programs under the auspices of DOE HQ.

The major actions of testing and experimentation within this work plan, to which this QAA apply, include:

- laboratory-scale steam extraction testing, to select optimal chelator-redox systems; and,
- bench-scale soil column washing tests, to evaluate leaching/mobilization of Pu/Am species with respect to the chelator-redox systems referenced above.

3.0 BASIS FOR TECHNICAL ACTIVITY

This work indirectly supports legally binding requirements stated in the Interagency Agreement (IAG) regarding mitigation and/or remediation of contamination at the RFP, especially soils within OU-2 and potentially OU-9. The work specifically supports Task 2 of the EM-50 funded Technical Task Plan (TTP) for Plutonium in Soils Integrated Demonstration Sampling Support.

4.0 BASIS OF QA REQUIREMENTS

The QAPjP was prepared to identify the QA requirements and methods applicable to the RFP Environmental Restoration (ER) Program activities, as identified in the Attachment 2 of the IAG Statement of Work. Section IV.A of the IAG specifies the minimum quality elements that the QAPjP must include, and references EPA QAMS/005/80, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, for guidance in preparing the QAPjP.

5.0 QUALITY REQUIREMENTS

5.1 Organization and Responsibilities

The DOE/RFO is responsible for the overall coordination of the Chemically Enhanced Steam Stripping Treatability Study due to the major organizations involved with this work, including LANL, LATO, and EG&G Rocky Flats.

An organization chart is shown in Figure 1. The organization has been structured such that quality is the responsibility of those who have been assigned the responsibility of performing the work, and conformance to established requirements is verified by individuals and groups not directly responsible for performing the work. The EG&G Rocky Flats Environmental Restoration Management (ERM) organization, specifically the Environmental Science and Engineering (ES&E) department, is responsible for management and coordination of the EG&G Rocky Flats resources dedicated to the project.

Responsibilities are as follows:

RFO Technical Program Officer - Provide contract oversight and update DOE/RFO on the progress of the project. Provide a communication link between the Agencies (CDH and EPA) and EG&G.

LATO Technical Project Manager - Provide a communication link between the RFO and EG&G Technical Project Managers with the LANL Technical Project Manager. Facilitate data transfer between RFP and LANL.

LANL Technical Project Manager - Work with LANL procurement to facilitate contractual matters. Assist the RFO and EG&G Technical Project Managers with communications with LANL personnel. Responsible for project management at LANL.

LANL Principal Investigator - Plans and directs the efforts of lab technicians performing the work.

LANL Technical Services - Perform CESS test work, complete the detailed test plan, write monthly, semi-annual, and annual reports.

LANL Analytical Services - Perform analytical lab work as deemed necessary by the work plan, determine the amounts of contaminants before and after performing the CESS tests.

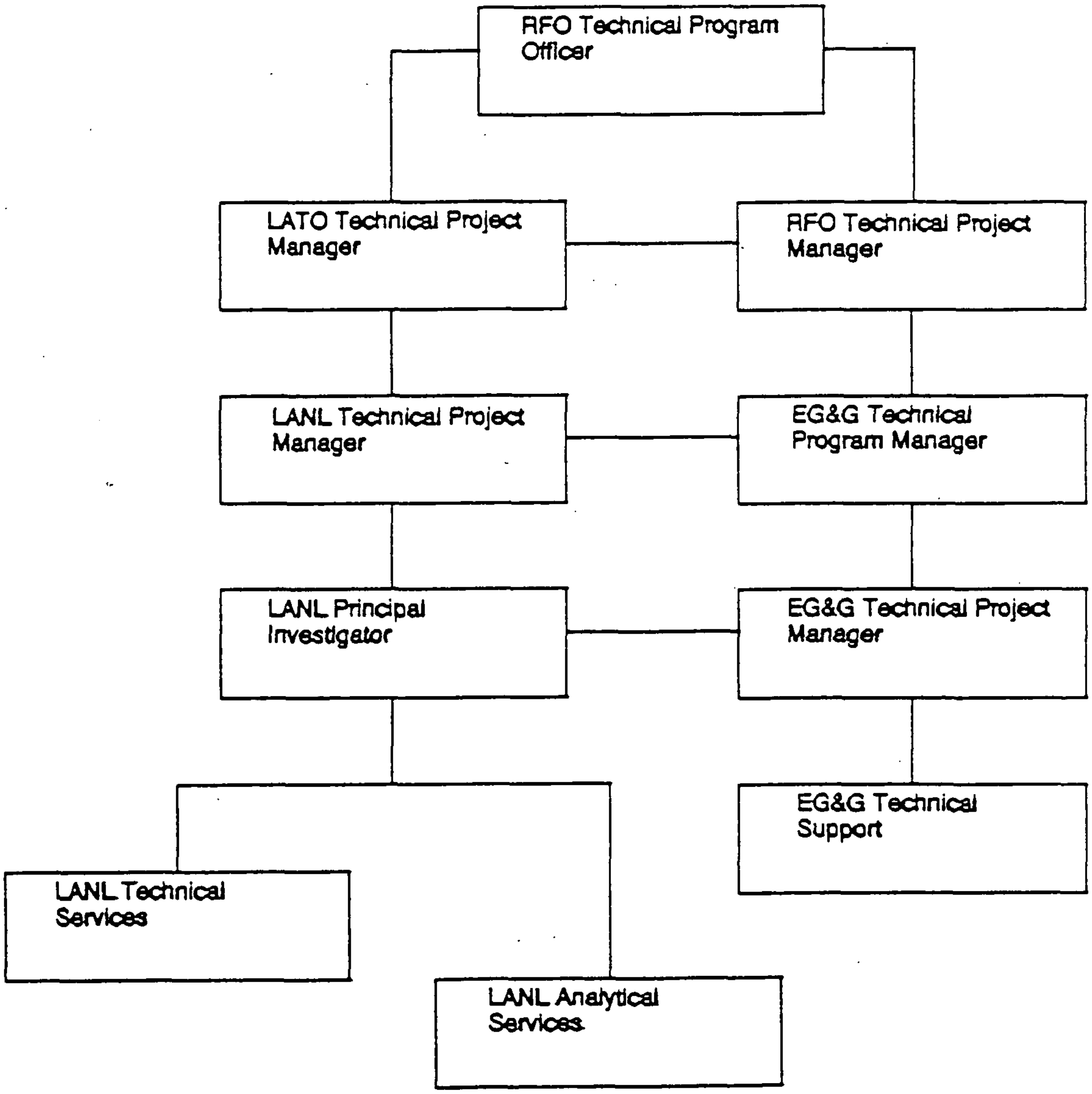
RFO Technical Project Manager - Facilitate procurement actions in DOE/RFO and provide budget and contract oversight.

EG&G Technical Program Manager - Provide contract oversight to monitor the progress of the project. Provide a communication link with DOE/RFO Technical Program Officer and the LATO Technical Project Manager.

EG&G Technical Project Manager - Provide oversight of the test work to monitor progress and quality. Facilitate procurement actions within EG&G and RFO. Ensure that budgetary and schedule constraints are followed.

EG&G Technical Support - Provide technical support for LANL Technical Services and the EG&G Technical Project Manager.

Figure 5-1. Management and Reporting Structure



5.2 Quality Assurance Program

The ERM Environmental Quality Support Division is responsible for providing internal quality implementation support (including inspections and surveillance of system acceptance and performance) to assure that the quality requirements of this QAA and the QAPjP are being implemented. LANL will work to the Quality Assurance Plan specific to Los Alamos and is subject to established and approved DOE Albuquerque Office (AO) Quality Assurance programs.

All EG&G Rocky Flats and subcontractor personnel that perform quality affecting activities on this project shall have qualification records that document they are qualified to perform their assigned tasks. The EG&G TD Manager shall identify any Rocky Flats Plant area-specific and/or specialized training requirements that are applicable to project personnel. Job specific training will include theory of operations, system components, principles of operations, system interrelationships, protective devices, and practical factors.

LANL personnel are required to complete all training required by the DOE-AO to be qualified to perform quality affecting activities on this project at Los Alamos. Training and qualification records must be maintained by LANL personnel to document that they are qualified to perform their assigned tasks.

LANL procedures and documents should be available for review by EG&G EQS and/or DOE at any time.

5.3 Design Control and Control of Scientific Investigations

The QAPjP considers activities that generate analytical data, which requires collection and analysis of environmental samples, to be scientific investigations. Controls for scientific investigations include developing data quality objectives, collecting and analyzing samples according to approved procedures, establishing and implementing quality controls, and reducing and reporting data in a controlled manner.

Established procedures shall be used for gathering samples in the field and subsequent testing in the laboratory(s). When deviations from the operations procedures occur, or when new or nonstandard procedures are implemented, a Scientific Notebook System (SNS) will be used as the primary means of documenting quality-affecting information.

Data quality objectives (DQOs) quantitatively and qualitatively describe the uncertainty that decision makers are willing to accept in results derived from environmental data (see Sections 5.12 and 5.13). This uncertainty is used to specify the quality of the data required to meet the objectives of the investigations. The process for developing DQOs for remedial investigations is summarized in Appendix A of the QAPjP. Precision, accuracy, representativeness, completeness, and comparability (referred to as PARCC parameters) are fundamental parameters used to indicate data quality.

Precision and accuracy are dependent on the analyte of interest, the sample matrix, analytical method, and the quality control procedures applicable to the method of analysis. Precision and accuracy of all measurements used to calculate results, as documented in the final report, shall be documented, as well as the physical/chemical methods used to produce the cited performance specifications (per item or system, as applicable).

Completeness can also be a quantitative measure of both the sampling and analysis process. However, because this project is based only on representative samples within the laboratory/bench scale settings, and not on a comprehensive field sampling scheme regarding site characterization, "completeness" in its typical context, is not an applicable quality indicator in this work plan.

Comparability and representativeness are qualitative parameters that are ensured through careful development of and adherence to sampling and analysis plans and procedures. Deviations from established sampling and analysis protocols, and potential impacts to data quality, shall be documented in the SNS. Samples sent to the laboratory(s) for testing shall represent physical and chemical characteristics of the soils to be potentially remediated. For comparability purposes, similarities and differences in the soil samples must be discussed in the final report relative to explanation of the test results.

A more detailed description of the PARCC parameters is included in Section 5.12.

5.4 Document Control

Documents produced by EG&G that control the work described in this work plan shall be controlled to ensure that key project personnel receive accurate and up-to-date information. Such documents shall be controlled per EG&G procedure 3-21000-ADM-5.01, "Document Control".

LANL shall acknowledge receipt of, and manage, EG&G plans and procedures in accordance with EG&G procedure 2-11000-ADM-06.01.

5.5 Control of Purchased Items and Services

Items or services procured under this project shall be performed in accordance with the requirements of the QAPjP and ER administrative procedure ADM-4.01, "Procurement Document Control", including retention of purchase order receipts, contracts, or any other documentation related to the integrity/traceability of the purchased product or service.

5.6 Inspection and Assessment

Quality affecting activities are subject to assessments and inspections. These assessments will be performed formally, in accordance with EG&G procedures (e.g., 3-21000-ADM-10.01 and/or -ADM18.02), or informally, as requested by line management. The work place and working records shall be accessible during normal working hours for verification or ERM internal assessments and inspections by EG&G or their representatives during the performance of this project. Any nonconformances identified during formal assessments shall be documented with Nonconformance Reports. Independent audits of the project may be conducted by the SAA organization in accordance with SAA procedures.

ERM internal assessments and inspections shall be performed by the EG&G Technical Program Manager or designee. Frequency of these audits will be determined after contract award. Additionally, EG&G Environmental Quality Support Division from Environmental Restoration shall review and approve Quality Assurance elements specific to this Work Plan and Statement of Work prior to contract award.

5.7 Sampling Procedures and Custody

A sample chain-of-custody (COC) will be initiated at the time the samples are collected and maintained through all transfers of custody until the sample is received at the testing laboratory. Procedure 5-21000-OPS.FO.13 provides instructions for preparing COC forms, and defines the procedures addressing sample containers, preservatives, handling, packaging, and shipping of soil samples collected at RFP. Samples shall be logged in upon receipt at the analytical laboratory and sample tracking throughout the analytical process shall be maintained in accordance with laboratory procedures.

5.8 Measuring and Test Equipment

Measuring and test equipment (M&TE) used in the construction/installation, inspection, and testing of the simulated in situ rad chemistry remediation system shall be selected, identified, calibrated, and maintained in accordance with the methods established in RFP administrative procedure 1-50000-ADM-12.01, Control of Measuring and Test Equipment. The M&TE requirements of Section 12 of the QAPjP are implemented through procedures specific to the sampling/analysis event, manufacturers instructions, and specific laboratory procedures.

5.9 Control of Nonconformances

Items, samples, and data that do not conform to specifications and/or requirements shall be identified, segregated (where necessary to prevent inadvertent use), dispositioned, and evaluated in accordance with approved procedures. Nonconformances related to the design, construction/installation, or testing of the testing system, and any waste related nonconformance, shall be controlled in accordance with RFP procedure 1-50000-ADM-15.01, "Control of Nonconforming Items, Samples, and Data".

5.10 Corrective Action

The identification, reporting, closeout, and documentation of significant conditions adverse to quality shall be accomplished in accordance with RFP procedures 1-50000-16.16, Corrective Action Program.

5.11 Quality Assurance Records

Project records that are considered ERM QA records include, but are not necessarily limited to, the final report, (including all appendices), design documents, procurement documents, construction/installation records, supplier/subcontractor evaluations, inspection records, test records, logbooks, sampling records, sample chain-of-custody records, analytical data packages, interim and annual operating reports, action plans, operation manuals, NCRs, CARs, audit reports, surveillance reports, self-assessment reports, personnel training and qualification records, the QAPjP, any administrative and procedures referenced herein, and any other project records that are used to support observations and conclusions in the final report. All ERM QA records generated shall be submitted to the ERM Records Center for processing according to ERM procedures 3-21000-ADM-17.01 and 3-21000-ADM-17.02.

5.12 Data Quality Objectives

The primary objective of this demonstration is to evaluate the effectiveness of the CESS technology in removing radionuclides from contaminated RFP soil. QA objectives are developed to produce data that can be used to evaluate the effectiveness of the technology. The following sections discuss topics directly related to the QA objectives. These include: data required; PARCC objectives, alpha detection levels.

5.12.1 Data Required

Data Uses:

- The data from the CESS Treatability Study will be used to determine if the technology efficiently extracts Americium and Plutonium from RFP soil.
- The CESS technology Treatability Study is being evaluated as an option for cleanup.

Data Types:

- Surface soil (<1/2") will be sampled in at least five five gallon pails at approximately 80 lbs per pail.
- Approximately 400 lbs of soil will be sampled and shipped to Los Alamos.
- Radionuclide analysis will be performed (using Alpha Spectrometry) for Am^{241} and $Pu^{239,240}$.
- The data generated by this treatability study will be used to assess the efficiency of the CESS technology. Thus, an 80% confidence level is acceptable for this "proof of principle" study, and large errors are acceptable as the efficiency approaches zero.

Data Quality Needs:

- Sampling Needs:
  - o Appropriate Analytical Levels.
    - To adequately test the removal efficiency of the CESS process, the soil must initially have activity of at least 1 nCi/g.
  - o Contaminants of Concern.
    - Americium
    - Plutonium
  - o Preliminary Remediation Goal
    - $Pu^{239,240}$ = 3.5 pCi/g
    - Am^{241} = 2.4 pCi/g
  - o Critical Samples
    - At least five five gallon pails at approximately 80 lbs per pail.
    - Approximately 400 lbs is necessary to perform the experiments, allowing for additional tests to be run in the event of experimental error and/or uncertainty.
    - Grain size must be <1/2". Plutonium and Americium have historically been found attached to smaller particulates in soil.

- Analytical methods for parameters of solid samples are shown in table 5-1.

Table 5-1. CESS Treatability Study QA objectives for Noncritical Measurements Solid Samples

Measurement	Method	Measurement Unit	Practical Quantitation Limits	Precision (RPD)[a]	Accuracy (% Recovery)	Completeness (%)
Radionuclides						
Plutonium 239, 240	EPA-600/7-79-081[b]/HEA-0018-01[c]	pCi/g	3.5	30	80-120	90
Americium 241	EPA-600/7-79-081[b]/HEA-0018-01[c]	pCi/g	2.4	30	80-120	90

Notes:

[a] RPD = Relative Percent Difference.

[b] Acid Dissolution Method for Analysis of Plutonium in Soils, U.S. EPA Environmental Monitoring and Support Laboratory, Las Vegas, Nevada, 1979.

[c] Maximum Sensitivity Procedures for Isolation of Plutonium and Americium in Composited Water Samples, Rocky Flats Plant Health and Safety Laboratories, Golden, Colorado, 1990.

5.12.2 PARCC Parameters

5.12.2.1 Precision

Precision is the degree of mutual agreement among individual measurements of the same property under prescribed similar conditions. Precision is evaluated by collecting and analyzing laboratory replicate samples.

For the measurements of plutonium and americium in solids and liquids, precision is determined by analysis of laboratory duplicate samples and calculating the relative percent difference (RPD). RPD is calculated using equation 5-1:

$$\% \, RPD = \frac{|A - B|}{(A + B)/2} \times 100 \, \% \qquad (5\text{-}1)$$

where:

% RPD	=	relative percent difference
A	=	first replicate concentration
B	=	second replicate concentration.

5.12.2.2 Accuracy

Accuracy is the degree of agreement between an analytical measurement and a reference accepted as true value. The accuracy of a measurement system is affected by errors introduced through the sampling process, field contamination, handling, sample matrix, sample preparation, and analytical techniques. Accuracy is evaluated through the use of standard reference materials (SRMs), QC check samples, calibration standards, sampling equipment rinsate blanks, and bottle rinsate samples.

Accuracy for radionuclides in solids will be estimated by comparing the true to the measured analyte level from a QC check sample using equation 5-2:

$$Accuracy = \frac{MSC}{TAC} \times 100 \tag{5-2}$$

where:

MSC = measured concentration in the QC check sample.
TAC = true analyte concentration in the QC check sample

5.12.2.3 Representativeness

For this project, representativeness involves sample size, sample volume, sampling times, and sampling locations. The QA goal is to obtain a statistically adequate number of samples that represent the various process matrices at the time that the samples were collected. The volume of samples collected also depends on the analytical method chosen, allowing for QC sample analyses and reanalysis, if needed.

Representative sampling of bulk soils for radionuclide analyses is difficult to achieve without some pre-treatment by physical methods. Reproducibility of analytical methods is also complicated by heterogeneities in the soil matrix. To minimize this experimental difficulty and maximize information specific to the chemical/steam conditions, soils will be dried and sieved prior to all bench-scale experiments. As with previous studies (where plutonium is associated with a particular size fraction in the RFP soils), a soil size fraction will be selected for bench-scale testing to optimize experimental logistics and performance, while maintaining sampling and analytical reproducibility. Additionally, all tests will be run in duplicate to ensure representativeness and data quality.

There will be 18 batch desorption experiments, all performed in duplicate. The Bench-Scale Column Experiments will involve eluting ten to twenty column volumes in duplicate. The Bench-Scale Optimization Column Studies will also be performed in duplicate, however the number of samples to be run will be determined during testing, since they involve optimizing leaching solutions. For each of these experimental stages, duplicate samples will be taken before and after treatment.

5.12.2.4 Completeness

Completeness is a measure of the percentage of project-specified data that is valid. Valid data are obtained when: (1) samples are collected in accordance with the EMD Manual Operations Procedure, "Environmental Sample Radioactivity Content Screening," (5-21000-OPS-FO.18, Rev. 1); and (2) none of the QC criteria that affect data quality are exceeded. The project completeness value will be calculated by dividing the number of valid sample results by the total number of sample analyses completed for this treatability study as shown in equation 5-4:
where:

$$\%C = \frac{V}{T} \times 100 \qquad (5\text{-}3)$$

% C	=	percent completeness
V	=	number of measurements judged valid
T	=	total number of measurements.

5.12.2.5 Ensuring Representativeness and Completeness

Representative sampling of bulk soils for radionuclide analyses is difficult to achieve without some pre-treatment by physical methods. Reproducibility of analytical methods is also complicated by heterogeneities in the soil matrix. To minimize this experimental difficulty and maximize information specific to the chemical/steam conditions, soils will be dried and sieved prior to all bench-scale experiments. As with previous studies (where plutonium is associated with a particular size fraction in the RFP soils), a soil size fraction will be selected for bench-scale testing to optimize experimental logistics and performance, while maintaining sampling and analytical reproducibility. Additionally, all tests will be run in duplicate to ensure representativeness and data quality.

There will be 18 batch desorption experiments, all performed in duplicate. The Bench-Scale Column Experiments will involve eluting ten to twenty column volumes in duplicate. The Bench-Scale Optimization Column Studies will also be performed in duplicate, however the number of samples to be run will be determined during testing, since they involve optimization of leaching solutions. For each of these experimental stages, duplicate samples will be taken before and after treatment.

5.12.2.6 Comparability

The comparability of the data will be minimized by using standard U.S. EPA analytical methods and by reporting data in a tabular or graphical format. All methods used will be specified and any deviations from methods will be documented, and all results will be reported in standard units as shown in Table 5-1. All laboratory calibrations will be performed with standards traceable to NIST or other EPA-approved sources. Alpha Spectrometry will be used to analyze the Pu and Am content in the soil after treatment. All samples will be analyzed in the same laboratory using the same analytical techniques.

5.13 Internal Quality Control Checks

An internal QC system is a set of routing internal procedures for verifying that the data output of a measurement system meets prescribed criteria for data quality. This system contains methods for measuring and defining the quality of the data output. LANL personnel performing the laboratory analyses will use the following internal analytical laboratory QC measures, where appropriate, to verify that the precision and accuracy objectives are met. The control limits for the critical parameters are listed in section 5.12. When these limits for the critical parameters are exceeded, the EG&G project and QA managers will be contacted.

Table 5-2 summarizes calibration and QC of analytical measurement equipment for specific analytical methods. QC checks consist of laboratory QC and field QC.

Table 5-2. CESS Treatability Study Calibration and Scheduled QC

Parameter/Method	Procedure	Frequency	Acceptance Criteria	Corrective Action
Plutonium, Americium HEA-0018-01	SRM	5 % or 1 per batch, whichever is more frequent	See Table 5-1	1) Evaluate other QC samples (blanks) in the batch, repeat analysis of sufficient sample 2) Recalibrate
	Method Blank	5 % or 1 per batch, whichever is more frequent	This criteria will be set during evaluation.	1) Assess source of contamination 2) Repeat Analysis 3) Flag Data 4) Inform EG&G project and QA managers

5.13.1 Laboratory QC

The QC checks to be performed in the laboratory consist of LCSs, replicate analyses, laboratory blank analyses, determining MDAs, chemical recovery criteria, aliquot sizes and other instrumentation checks.

5.13.1.1 LCSs

- LCSs shall be analyzed at a frequency of 5% per batch.
- LCSs shall be prepared and analyzed in the same manner as the samples.
- LCSs shall have the same aliquot size as the samples.
- LCSs shall have the same PRGs as the samples.
- Using the Alpha Spectrometry Overall Counting Uncertainty, the observed value of the LCSs shall be within 1σ control limits of the expected LCS value and have a relative percent error that does not exceed 50%.
- LCSs shall be counted for the same count durations as the samples.
- LCS data shall be submitted with each data package and shall include the expected values for all isotopes for which the samples are being analyzed.
- An LCS with a deionized water matrix may be used as an LCS for samples with matrices other than that of water.

5.13.1.2 Replicate Analyses

- Replicate analyses shall be analyzed at a frequency of 10% or one per batch.
- Replicate samples shall be prepared and analyzed in the same manner as the samples.
- Replicate samples shall have the same aliquot size as the samples.
- Replicate samples shall have the same PRGs as the samples.
- Replicate analyses data shall be submitted with each data package.
- The replicate analyses shall be within the 3σ range of the weighted average and its associated standard error. "Hot" particles may be present in soils, sediments, and total waters and this will be taken into consideration when evaluating duplicates.

5.13.1.3 Laboratory Blank Analyses

- Laboratory blanks shall be analyzed at a frequency of 5% or one per batch.
- Laboratory blanks shall be prepared and analyzed in the same manner as the samples.
- Laboratory blanks shall have the same aliquot size as the samples.
- Laboratory blanks shall be counted for the same count duration as the samples.
- Deionized water may be used as a laboratory blank for the samples.

5.13.1.4 Determining MDAs

- Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the PRGs. Interfaces, contaminants, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate that the MA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to matrix problems will be treated as an additional sample analysis. In all cases, MDAs which fail to achieve the required PRG shall be fully explained in the case narratives.
- The MDAs shall be reported on sample calculations sheet. The last background taken (1 month old or less) shall be used for calculations.

- The laboratories shall compile quarterly a history of RFP laboratory blanks used to perform the sample analyses. The analytical results shall be submitted on a quarterly basis.
- The MDA shall be calculated as shown in equation 5-5:

$$MDA\ (pCi/Aliquot\ in\ appropriate\ units) = \frac{4.65\ S_B + \frac{2.71}{TEY}}{a*Aliquot} \quad (5\text{-}5)$$

where:

S_B	=	Standard deviation of the population of quarterly RFP blank values (DPM)
T	=	Sample count duration in minutes
E	=	Detector efficiency
Aliquot	=	Aliquot in appropriate units
Y	=	Chemical recovery for the sample
a	=	2.22 conversion for Dams to pico Curies.

5.13.1.5 Chemical Recovery Criteria

- Chemical recovery for Pu and Am analyses shall be >20% but <105%. Chemical recoveries outside these limits require the affected samples to be reanalyzed.
- Chemical recovery shall be calculated based on the latest instrument efficiency value.
- Counts obtained for the tracer peak, Dams of tracer used, and aliquot of tracer used shall appear in the raw data.

5.13.1.6 Aliquot Sizes

- The aliquot size shall be optimized to achieve the PRGs. If the PRGs are not achieved and the aliquot sizes are less than 1.0 Liter for Am and Pu, then the problem shall be addressed in the Case Narrative.